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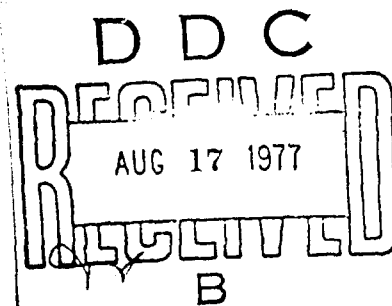
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BARRIER FILMS FOR MK 12 MOD 2 CARTRIDGE PLUG

Final Report
(23 July 1973 to 23 July 1974)

August 1974

By
N. W. Gregornik



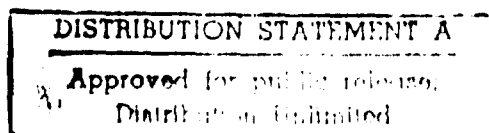
Prepared Under Contract N00174-74-C-0015

For

Naval Ordnance Station
Indianhead, Maryland
Department of the Navy

By

Honeywell Inc.
Government and Aeronautical Products Division
600 Second Street North
Hopkins, Minnesota 55343



UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R & D		
<small>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</small>		
1. ORIGINATING ACTIVITY (Corporate author) Honeywell Inc. Government and Aeronautical Products Division 600 Second Street North, Hopkins, Minnesota 55343		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED
		2b. GROUP
3. REPORT TITLE BARRIER FILMS FOR MK12 MOD 2 CARTRIDGE PLUG		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final (July 1973 through July 1974)		
5. AUTHOR(S) (First name, middle initial, last name) Gregornik, N. W.		
6. REPORT DATE August 1974	7a. TOTAL NO. OF PAGES 46	7b. NO. OF REFS 47
8a. CONTRACT OR GRANT NO. N00174-74-C-0015	8b. ORIGINATOR'S REPORT NUMBER(S)	
b. PROJECT NO.		
d.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10. DISTRIBUTION STATEMENT	DISTRIBUTION STATEMENT A Approved for public release; Distribution Unlimited	
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY / Naval Ordnance Station Department of the Navy Indianhead, Maryland	
13. ABSTRACT An investigation was conducted for the purpose of selecting a film barrier to minimize permeation of nitroglycerin vapors from M26 propellant into a polyurethane foam cartridge plug. This investigation consisted of a literature survey, polymer selection and screening, and 16 week permeation tests. The results of the study showed fluorinated ethylene propylene (FEP) to provide the best barrier against nitroglycerin vapors, but an interaction between this film and the M26 propellant was detected. The second best barrier was poly (chlorotrifluoroethylene) (CTFE). None of the films tested were found to be affected by the nitroglycerin vapors. Solubility parameter was found to be the most useful predictor of nitroglycerin permeation rates.		

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Barrier Films Nitroglycerin Vapors Permeation Rate Glass Transition Solubility Parameter Dielectric Constant Polarographic Analysis Thermal Analysis Compatibility Propellants Ammunition						

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SECTION 1 INTRODUCTION

The purpose of this study is to select a barrier material for use around the MK 12 Mod 2 plug. This selected barrier material should minimize or prevent migration of nitroglycerin and water vapor into the plug's material.

The MK 12 Mod 2 plug is a closed-cell, rigid polyurethane foam plug. This plug is used to cap and seal 5" / 54 cartridge cases. One of the propellants used in these 5" / 54 cartridge cases is a double-based propellant containing nitroglycerin, $C_3H_5(NO_3)_3$. If the plug absorbs nitroglycerin or water, four problems may occur:

- Creation of explosive hazard - The nitroglycerin may concentrate in the plug.
- Degradation of the MK 12 Mod 2 plug's physical properties - The nitroglycerin may break down this plug's chemical structure.
- Change in physical dimensions - Absorption of atmospheric water vapors during storage will increase the plug's dimensions. Such an increase could prevent this plug's insertion into the 5" / 54 cartridge case.
- Loss of nitroglycerin may change the propellant's performance.

SECTION II BACKGROUND DISCUSSION

Prior to selecting the barrier material, members of Honeywell's Plastics Laboratory conducted a literature search and study on nitrated esters through polymers. (Nitrated esters are nitroglycerin and related compounds.) First, members of the Plastic Laboratory sent a request to the Defense Document Center (DDC) seeking information on the permeation of nitrated esters. Results of this search were negative. Next, Honeywell's Engineering Library conducted a search with a much broader scope. Plastics Lab members prepared and reviewed a bibliography on plastics permeability. They extracted and ordered the most promising references from this bibliography. (Appendix I contains a reference listing.)

To select materials, a specific set of guidelines and criteria had to be established. Processing and permeability standards were chosen as selection criteria.

Processing is the method of inserting the polymer barrier between the vapors and the plug. Within all practical constraints; injection molding, blow molding, vacuum forming, injection molding/heat shrinking (cross-linked thermoplastic), and spray or dip coating are applicable processes (Figures 1 - 5).

Permeation is the process of a molecule dissolving into the polymer at one surface and winding through the polymer chains to the opposite surface and finally evaporating. A detailed discussion of the mathematical theories of permeation has been summarized. (See reference 18, Appendix I for more details.)

Permeability is the product of solubility and diffusivity, i.e.:

$$P = SD$$

where

P = Permeability

S = Solubility constant (Henry's Law)

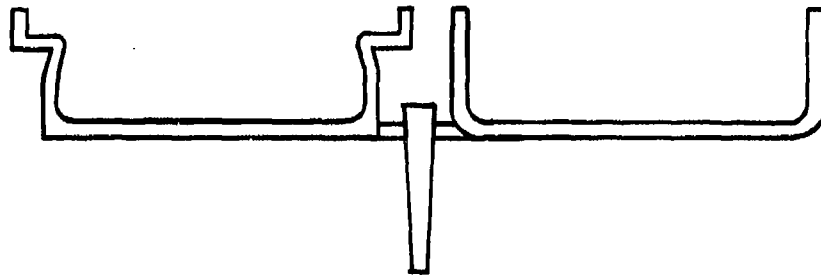
D = Diffusivity constant (Fick's Law)

Note: S and D obey Arrhenius relationship

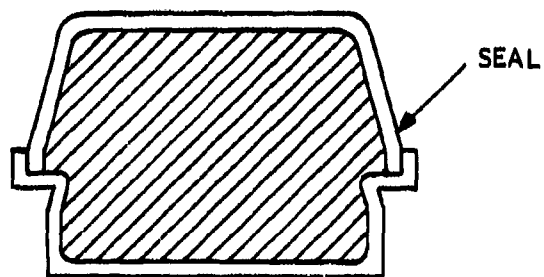
⇒ P follows Arrhenius relationship.

$$P = P_0 \exp (E_p/RT)$$

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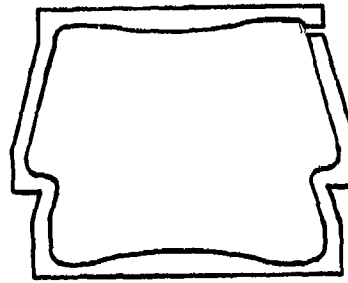


STEP 1. MOLD ASSEMBLY

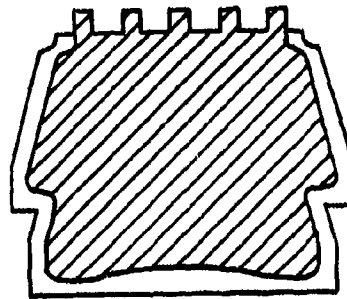


STEP 2. INSERT PLUG AND SEAL

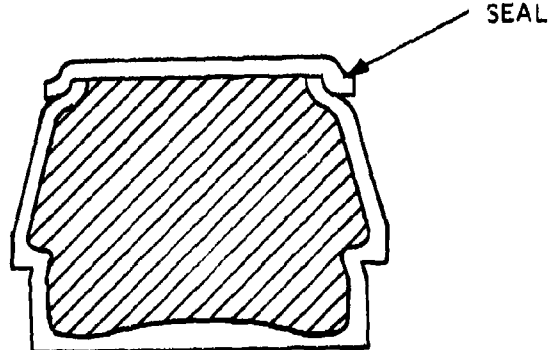
Figure 1. Injection Molding Process



STEP 1. MOLD PART

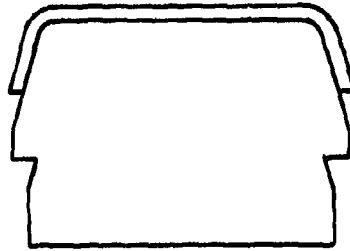


STEP 2. TRIM AND FOAM PLUG IN PART

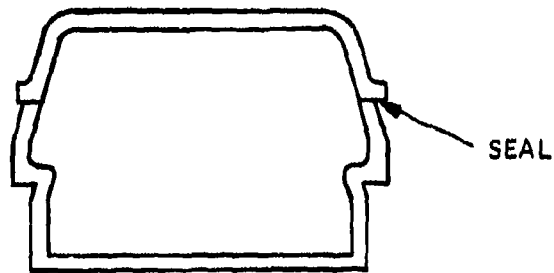


STEP 3. CAP AND SEAL

Figure 2. Blow Molding Process

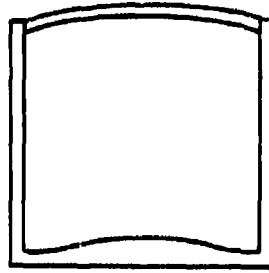


STEP 1. FORM BOTTOM ONTO PLUG AND TRIM



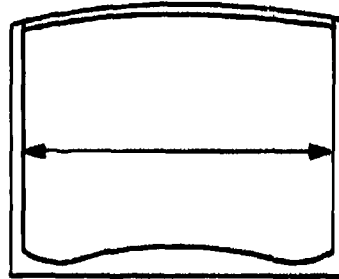
STEP 2. FORM BASE ONTO PLUG, TRIM AND SEAL

Figure 3. Vacuum Forming Process

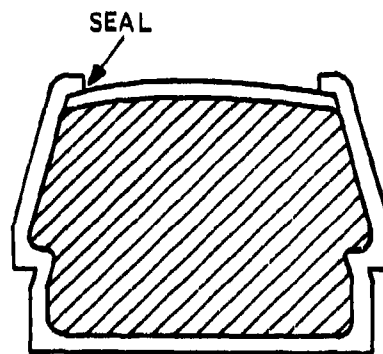


STEP 1. MOLD PART

STEP 2. IRRADIATE (CROSSLINK)

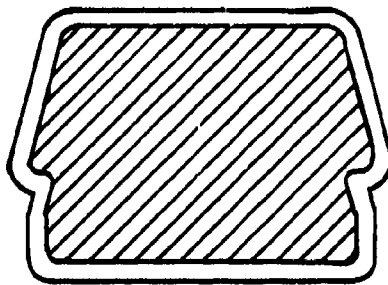


STEP 3. EXPAND

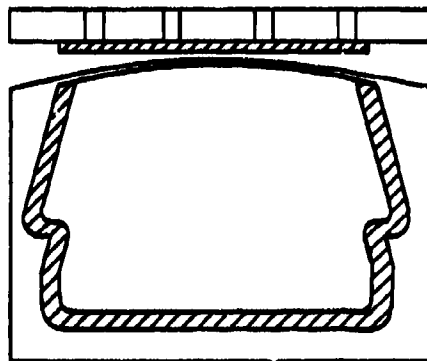


STEP 4. INSTALL PLUG AND CAP
SHRINK AND SEAL

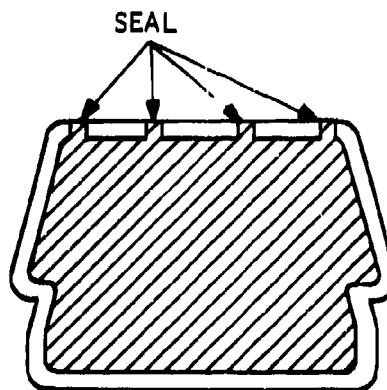
Figure 4. Injection Molding/Heat Shrinking Process



STEP 1. SPRAY OR DIP COATING ONTO PLUG



STEP 1a. COAT INTERIOR MOLD SURFACES



STEP 2a. FOAM PLUG AND CURE, REMOVE AND SEAL VENTS

Figure 5. Coating Process

where

E_p = Activation energy of permeation and the sum of $(H + E_d)$

H = Heat of solution

E_d = Activation energy of diffusion

R = Gas constant

T = Absolute temperature

(Refer to Appendix I for source listing).

Control of permeation for this study would have to remain solely within material choice. Other factors affecting permeation; temperature, concentration gradient, and pressure cannot be changed or controlled. Analysis of published results of polymer properties affecting permeation are discussed below.

DIFFUSION

Factors affecting diffusion are:

- Crystallinity -- A crystalline polymer contains crystallites within an amorphous matrix. Scanning electron microscopy has shown crystallites to be composed of aligned and tightly folded portions of polymer chains, Figure 6. Therefore, the only path left for permeation is through the amorphous regions. Crystallinity also retards permeation by decreasing the solubility of the polymer.
- Cross-linking -- A cross-linked polymer contains chains of the polymer attached to the other chains by covalent bonds, Figure 7. These connections may be relatively close and in high quantities per chain (tight network) or far apart and few in number (loose network). The effect of cross-linking is to decrease mobility of the chain segments. Very light cross-linking tends to increase the permeability of crystalline polymers by hindering the formation of crystallites. Amorphous polymers cross-linked loosely or tightly and crystalline polymers tightly cross-linked exhibit lower permeability.
- Transition Points -- It has been stated earlier that the property of permeation obeys the Arrhenius relationship with temperature. The exception to this rule is found at first and second order transitions. As the second order transition (T_g)

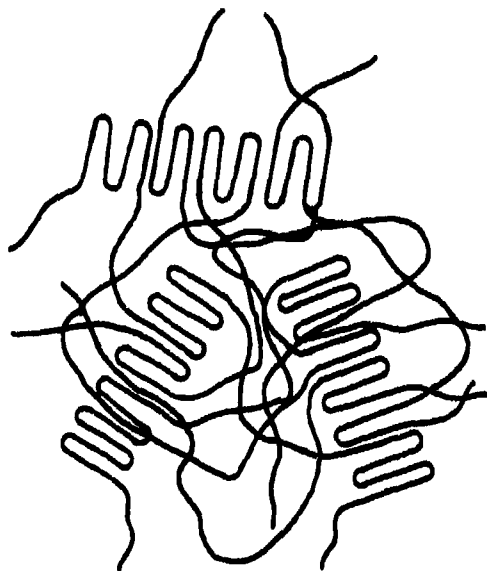


Figure 6. Model Showing Lamellae Tied Together
by Interlamellar Amorphous Chains

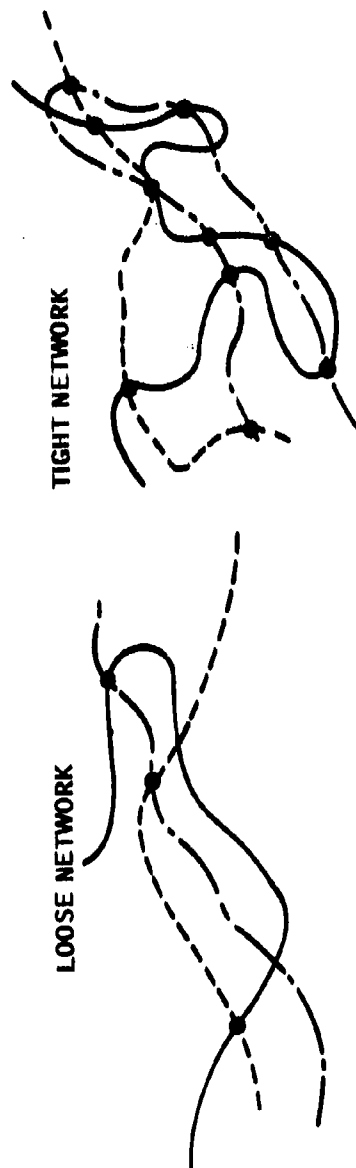


Figure 7. Cross-linking

is approached, molecular motion (chain flexing) begins and the rate of change of permeation increases markedly, Figure 8.

SOLUBILITY

Factors affecting solubility are:

- Solubility Parameter -- This parameter is the square root of cohesive energy density. It is a function of the molecular attraction forces. Liquids with like solubility parameters tend to dissolve like solutes and be miscible (assume no reaction). On a qualitative basis, solvents with solubility parameters widely divergent from a polymer will be least soluble in the polymer.
- Polarity -- The effect of polarity is akin to the solubility parameter in that like tends to dissolve like. Polar dissolves polar and non-polar dissolves non-polar and the effect on the solubility constant is the same as with similar solubility parameters.
- Hydrogen Bonding -- The ability of a material to form hydrogen bonds is shown by its hydrogen bonding index. The effect of a high-index solvent combined with a polymer containing a large quantity of hydrogen with which to form bonds increases the solubility index and, therefore, the permeation.

"IDEAL" POLYMER

The literature search did not result in data retrieval specifically applicable to the problem. Therefore, it was necessary to select material candidates using other than direct comparisons. The method chosen for selection was to:

- Evaluate and characterize the permeants.
- Describe an "ideal" polymer which will have the best chance of defeating permeation.
- Select and screen polymers which most closely match the properties of the "ideal" polymer.

The permeants in this case are nitroglycerin and water. Table 1 lists their properties which affect selection of candidate materials. As shown, the solubility parameters are widely different. If an arbitrary factor of ± 2.0 is chosen as the safety margin governing polymer selection, the solubility parameter must then fall outside the boundaries of

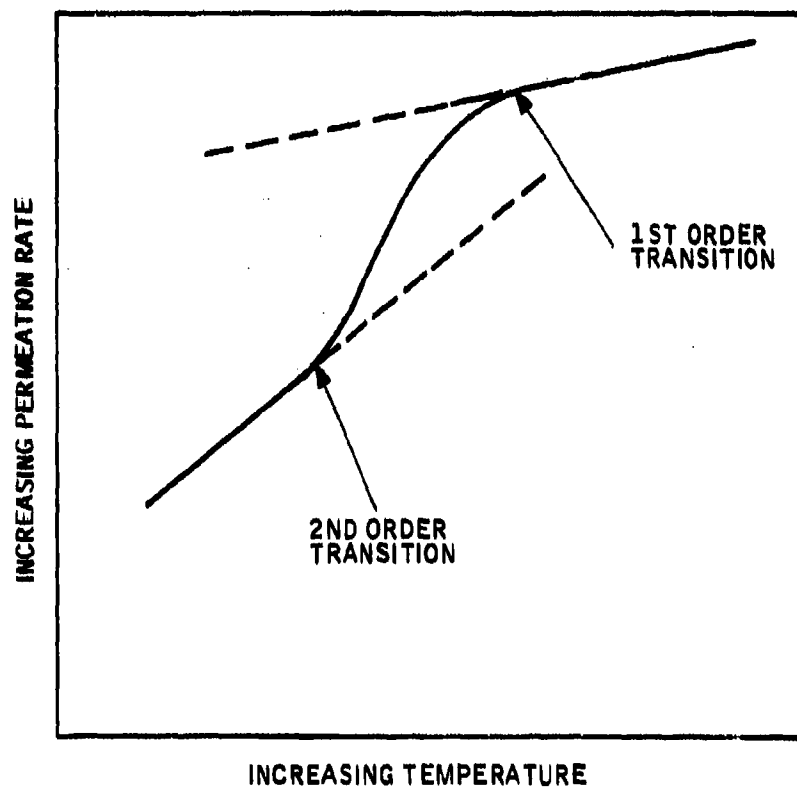


Figure 8. Transition Points

13.2 to 17.2 and 21.4 to 25.4. Secondly, since both permeants are polar (dipole moment > 0), a non-polar film is dictated by this criteria (dielectric constant = 0). Hydrogen bonding will be a factor affecting the permeation of water only since the availability of active hydrogen-bonding sites are minimal in nitroglycerin.

Other properties are not directly associated with permeant characteristics but of a more general nature are:

- 1) Glass transition (T_g) should be above the normal use temperature. Normal storage conditions generally are considered to be -65°F to $+160^\circ\text{F}$. Since nitroglycerin begins to degrade at 124°F (Note: Urbanski, 40), 122°F is the temperature chosen for polymer screening and aging.
- 2) The "ideal" polymer should be crystalline and/or cross-linked. The regularity of the chemical structure required for crystallinity will rule out random copolymers and branched polymers. Table 2 is a summary of properties considered necessary for the ideal polymer.

Table 1. Nitroglycerin and Water Properties

	Nitroglycerin	Water
Chemical Formula	$\text{C}_3\text{H}_5(\text{ONO}_2)_3$	H_2O
Solubility Parameter	15.2 ⁽⁴⁾	23.4 ⁽¹⁾
Dipole Moment	3.82 ⁽³⁾	1.84 ⁽²⁾
Hydrogen Bonding Index	Not Found - considerably lower than water ⁽⁵⁾	39.0 ⁽¹⁾

(1) Rodriguez (38)

(2) Lange (39)

(3) Urbanski (40)

(4) Calculated using the method of Fedors (31)

(5) Private communication Dr. S. Prager 2/7/74

Table 2. "Ideal" Polymer

Solubility Parameter (δ)	- 13.2 > δ > 15.2; 21.4 > δ > 25.4
Dielectric Constant	- 0
Glass Transition (T_g)	- Greater than 124° F
Crystalline	- Yes
Cross-linked	- Yes, if tight network
Polar	- No
Branched	- No
Copolymers	- No

SECTION III TECHNICAL DISCUSSION

Testing during this study was divided into two phases. Phase I consisted of selection and screening of polymer candidates. Phase II consisted of 16 weeks of testing and compatibility tests with the four most promising materials.

PHASE I (POLYMERS SELECTION AND SCREENING)

Using the description of the ideal polymer as a guideline, published data on plastics were researched and a list of likely candidates prepared. Table 3 lists the polymers selected for screening and the properties used for judging their applicability. After selection of the candidates, samples were ordered from their manufacturers and subjected to screening tests.

Screening the candidates was accomplished using two tests:

Test 1. Water vapor transmission was determined using a Honeywell Model W825 water vapor transmission rate tester. This unit, depicted in Figure 9, determines the rate of water vapor permeation through a test specimen by timing the change from 10% to 11% RH in the test chamber.

Test 2. The effect of nitroglycerin on the candidates was measured by a special test. The test chosen was an adaption of ASTM D 1460 - "Change in Length of Elastomers from Liquid Immersion." This test normally measures the change in length of a 100 mm long specimen caused by immersion in a fluid and mathematically relates this to the change in volume. This test was modified in the following manner. First, for safety considerations, a 0.01 M solution of nitroglycerin in ethanol was used instead of neat nitroglycerin. Results from this solution were then compared with results obtained by immersion in ethanol alone. Secondly, to minimize the amount of solution required for each test, the sample size was changed to 2.8 inches. Since the samples were selected based on their theoretical insolubility with nitroglycerin, length measurements were done with a shadowgraph capable of measuring to 0.0001 inch.

Table 3. Polymers Selected for Screening Tests

Polymer Name	Trade Name Tested	Source	Crystalline	Dielectric Constant	Parameter (6) (cal/cm ³ 0.5	Glass Transition (°C)	Applicable Processes
Acetal Copolymer	Celcon	Celaesee	Yes	3.7 ¹	Not Avail.	- 60 ⁴	A
Polybutylene	Butyl LM	Exxon Chem	No	2.3 ²	7.9 ²	- 73 ³	D
Chlorotrifluoroethylene	Aclar	Allied Chem	Yes	2.5 ¹	*	45 ²	A, B, C
Fluorinated ethylene-propylene	Teflon	DuPont	No	2.1 ¹	6.2 ²	-100 ²	A, B, C
Nitrile	Vicobar	DuPont	No	Not Avail.	15.0 ²	100 ³	C
High density polyethylene	Alathon	DuPont	Yes	2.3 ¹	8.0 ²	120 ²	A, B, C
poly (vinyl chloride)	Bakelite	Union Carbide	Yes	3.15 ¹	10.8 ²	81 ²	A, C
poly (vinyl fluoride)	Tedlar	DuPont	Yes	7.4 ¹	15.0 ³	40 ²	A, C
poly (vinylidene chloride)	Saran	Dow Chem	No	4.2 ¹	12.2 ²	- 19 ²	A, C
poly (vinylidene fluoride)	Kynar	Pennwalt	No	7.7 ¹	13.3 ³	- 45 ²	A, C
polyethylene terephthalate	Mylar	DuPont	No	3.2 ¹	10.7 ²	69 ²	C

Data Sources

- 1 Modern Plastics Encyclopedia (41)
- 2 Brandrup & Immergut (42)
- 3 Vendor
- 4 Billmeyer (43)
- 5 Roff & Scott (44)

* Not available, should be close to FEP and Tetrafluoroethylene (TFE) or approx. 6.2

Process Identification

- A. Injection Molding
- B. Heat Shrinking
- C. Vacuum Forming
- D. Coating

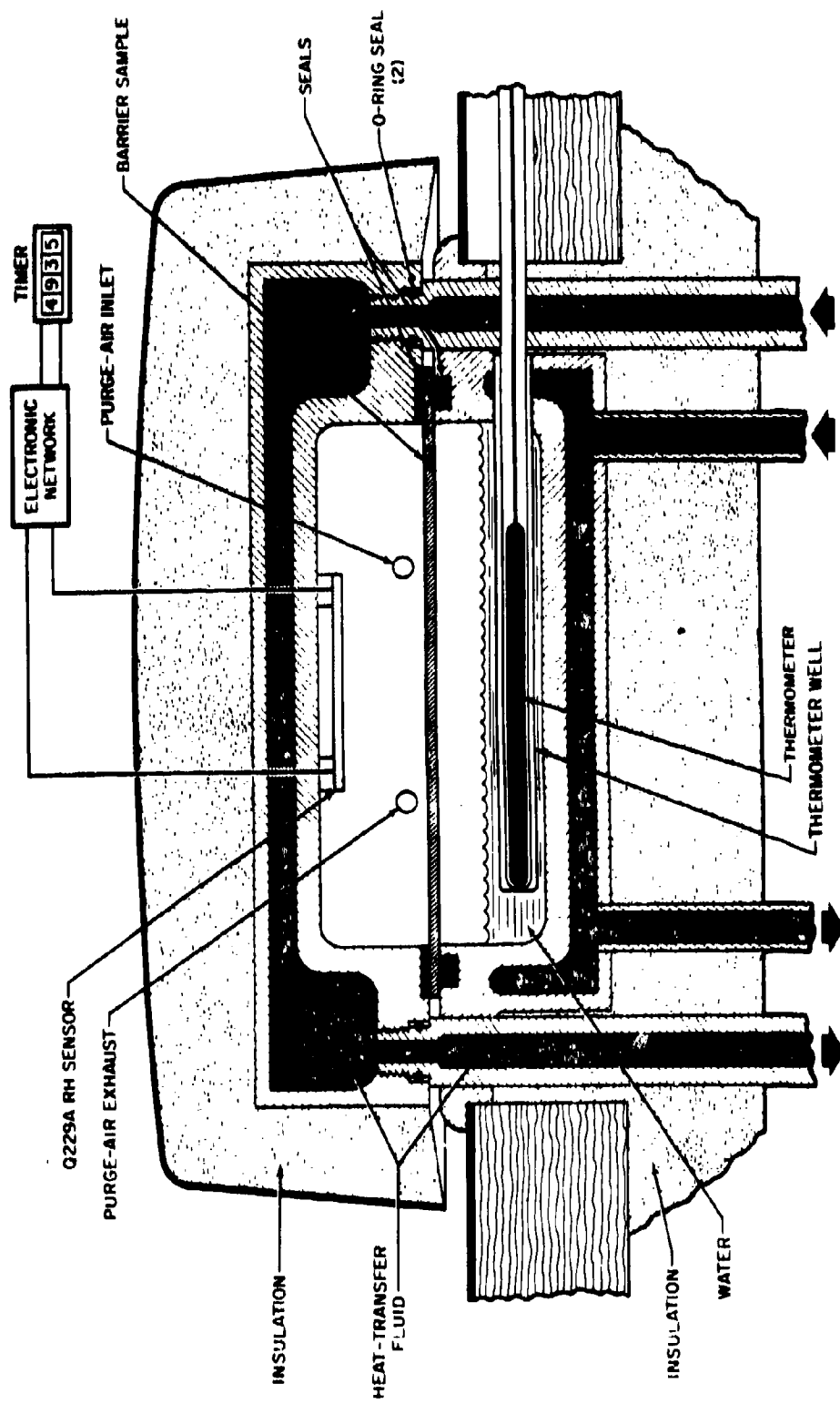


Figure 9. Sectional Diagram of Test Chamber

Appendix II is a detailed procedure for these tests and Table 4 depicts the results of testing. The four materials marked with an asterisk - Fluorinated ethylene - propylene (FEP), poly (Chlorotrifluoroethylene) (CTFE), Butyl coating LM73E66-18-0, and heat shrinkable cross-linked polyolefin RNF100 - were chosen for extended aging permeation tests.

PHASE II (PROLONGED PERMEATION TESTS)

At the beginning of this study, permeation cells were designed and manufactured. These cells, Figure 10a, were planned to simulate the conditions which would exist in the end use of the foam plug. Vapors emanating from the propellant in the bottom half of the cell, in order to escape, would have to pass through the material under test. Once vapors passed through this barrier it would be absorbed by the foam. The amount of nitroglycerin absorbed would then be determined by polarographic analysis.

Polarographic analysis is an electrochemical analytical technique used to determine the amount of specific ions in solution. For these tests, a dropping mercury anode is used to reduce the NO_2^- anion. The test is selective in that the potential at which oxidation (or reduction) occurs defines the reacting ion and the resulting current is directly proportional to the concentration of the ion in solution. Testing for this program was done on a Princeton Applied Research Model 174 polarograph. Nitroglycerin content of the unknown samples was determined by comparison with a standard (0.01M) solution of nitroglycerine in ethanol supplied by NOS/Indian Head.

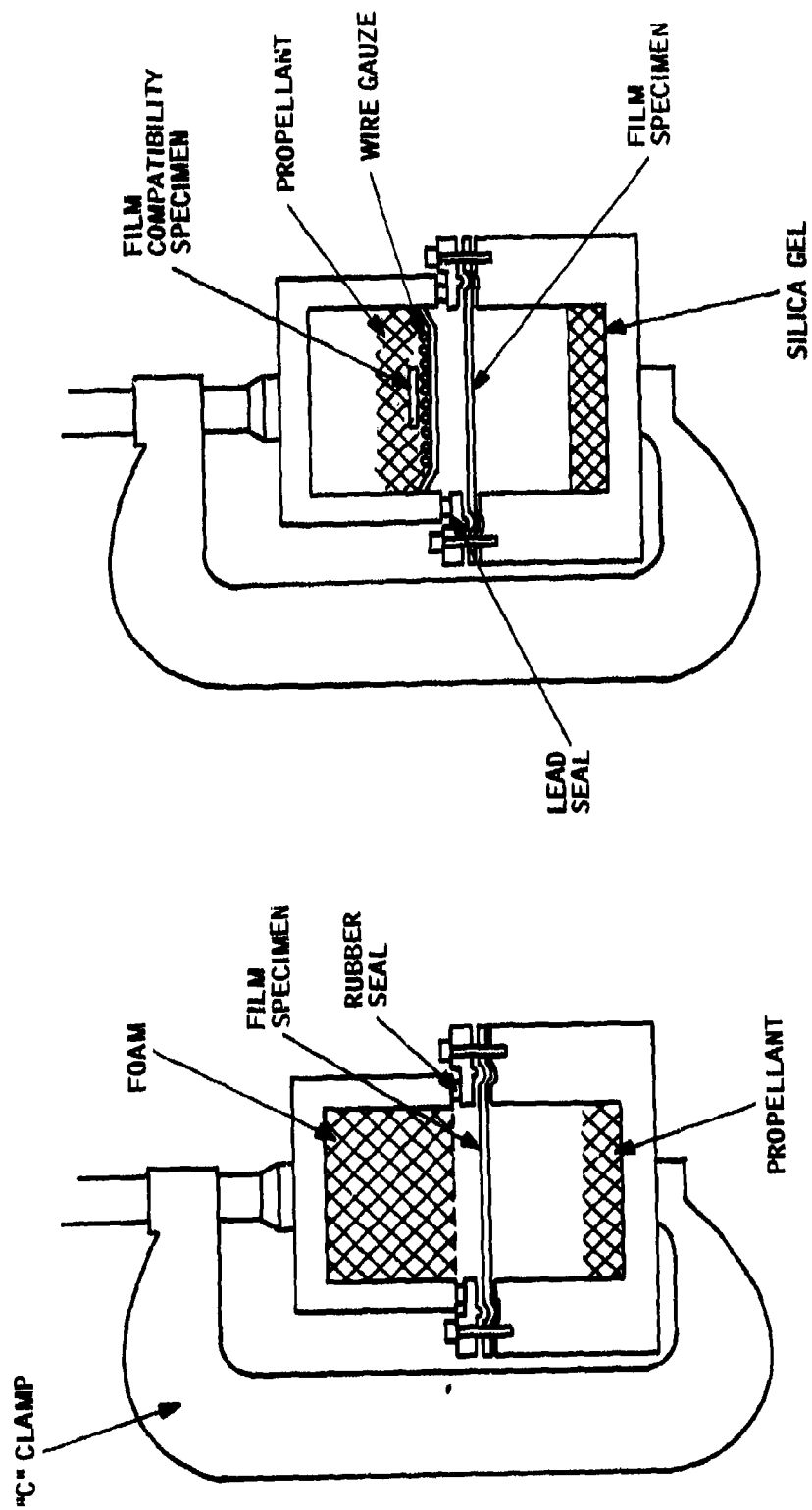
The concept of this procedure was tested by running the cells with foam alone, foam plus propellant, and foam plus propellant plus a film of low-density polyethylene. The cells were sealed and exposed to a temperature of 160° F for one week. These tests indicated two deficiencies. The first, absorbance of nitroglycerin by the rubber seal. This was corrected by substituting a lead seal. The second problem, however, was not as easily solved.

After the one-week aging period visible differences were apparent between the different test conditions. The foam tested by itself remained white. The foam with propellant and no film turned dark yellow, and the foam with film between it and the propellant turned light yellow. Attempts to determine the exact amount of permeation by polarographic analysis proved inadequate. Different samples of foam exposed to the same conditions

Table 4. Results of Screening Tests

Polymer	Designation	Gross Volume Change in .01M Nitroglycerin (percent)	Net Volume Change (Comparison with samples in ethanol) (percent)	Gross Weight Change in .01M Nitroglycerin	Water Vapor Transmission Rate (Grams/100 in ² 24 hr. - mil)
Acetal Copolymer	XF300	2.9	0.0	2.08	10.0
Polybutylene	73E66-16-0	4.7	0.50	6.42	0.16
Polybutylene	73E66-16-1	3.5	0.20	6.75	0.18
Polybutylene	73E66-16-2	4.7	Sample Distorted	3.9	0.27
* Polybutylene	73E66-18-0	0.7	-0.25	0.81	0.58
* CTFE	Aclar	0.32	0.14	0.02	0.04
* FEP	Teflon	-0.23	0.15	0.08	0.12
Nitrile	Vicobar	-0.22	-0.45	0.45	1.7
poly (vinyl chloride)	Bakelite VFA 3310	> 3.0	Sample Distorted	3.84	1.8
poly (vinyl fluoride)	Tedlar	2.7	0.38	1.02	1.2
poly (vinylidene chloride)	Saran	-7.0	0.20	-5.4	0.20
poly (vinylidene fluoride)	Kynar	4.3	-0.10	-0.17	2.1
polyethylene terephthalate	Mylar	2.4	-0.16	1.15	1.0
Olefins					
High density polyethylene		0.96	0.62	0.44	0.24
* Cross-linked polyolefin	RNF 100	0.20	-0.22	-0.08	0.38
Polyethylene/Saran laminate	Sarabex	0.38	-0.09	2.36	0.30

* Denotes materials selected for long term permeation tests



b. Final Configuration

a. Initial Configuration

Figure 10. Permeation Cell Design

yielded widely scattered results. Also, specimens tested on one day would show additional nitroglycerin leached into solution by the following day. This led to the conclusion that the nitroglycerin was somehow reacting chemically rather than being absorbed by the foam.

In an effort to resolve this problem, the following five alternates were tried:

- 1) Substituting activated carbon as the absorber and test as before.
- 2) Substituting silica gel as the absorber and test as before.
- 3) Running test as before, but digesting (chemically break-down) the foam prior to polarographic analysis.
- 4) Running test as before, but substituting bomb calorimetry for the polarographic analysis.
- 5) Running test as before, but substituting differential scanning calorimetry for the polarographic analysis.

Methods 1, 2, and 4 produced repeatable results while 3 and 5 did not. Polarographic analysis (1 and 2) was chosen over bomb calorimetry (5) because results could be obtained faster. Method 2 with silica gel as the absorber was chosen over Method 1 with activated carbon because of slightly greater amounts of nitroglycerin detected (see Table 5). This was thought to be the result of more efficient retrieval of the nitroglycerin from the silica gel.

Table 5. Barrier Film Cell Tests

Film	Absorber	Polarographic Analysis (ppm)
High Density Polyethylene	Activated Carbon	73.0
High Density Polyethylene	Silica Gel	74.7
Low Density Polyethylene	Activated Carbon	174.0
Low Density Polyethylene	Silica Gel	201.1

Appendix III is the test procedure used for the polarographic determination of nitroglycerin in the silica gel absorber.

After developing a satisfactory test and analysis procedure, prolonged permeation tests were begun. To ease handling of the test apparatus, the configuration of the cell set-up was changed by reversing the placement of the propellant and absorber. This set-up, Figure 10b, also allowed the replenishment of propellant in the upper chamber periodically

without disturbing or contaminating the absorber in the lower chamber. Also shown in the upper chamber is a piece of the film under test. The purpose of this specimen is to provide a sample of film exposed to the high nitroglycerin content in the upper chamber and test for possible deleterious effects on thermal properties (T_g) of the film.

The test itself consisted of setting up five cells of each film and placing them in an oven controlled at 122° F (Nitroglycerin will degrade at temperatures >122° F). At logarithmic intervals of 1, 2, 4, 8, and 16 weeks, one cell of each film was removed and tested. To ensure an adequate supply of nitroglycerin available for permeation, propellant was changed at 3, 6, 9, 12, and 15 weeks.

After disassembling the cells, the silica gel was analyzed for nitroglycerin content and the tensile strength and elongation of the film were tested per ASTM D 1708. Figures 11 - 13 depict the results of the tests. At the same time, the film in the upper chamber was tested via Thermo-Mechanical Analysis (TMA) on a Perkin-Elmer TMA apparatus in the penetration mode. This test will determine the T_g of the amorphous portion of a crystalline polymer. Shifting of these points during the aging test would indicate a change in polymer structure (e.g. degradation or cross-linking) or molecular arrangement (e.g. plasticization). Data from these tests are shown in Table 6.

Table 6. Effect of Aging on T_g (Softening Point) of Barriers

Film	T_g , °C					
	Control (No Aging)	1 Week	2 Weeks	4 Weeks	8 Weeks	16 Weeks
Teflon FEP	64	67	66	64	66	67
Aclar CTFE	47	68	56	74	69	67
Butyl LM	-58	-59	-59	-57	-58	- 58
X-Linked Olefin RNF100	99	99	100	89	90	102

COMPATIBILITY TESTING

To ensure that no deleterious effects might be caused by interaction between the film candidate and the propellant, compatibility testing was run concurrently with long-term permeation tests.

To check for effects of the plastic on the explosive, samples of the explosive were aged in an envelope of the plastic at the same temperature, time span, and withdrawal periods as the permeation tests, Appendix IV. After withdrawal, the detonation temperature of

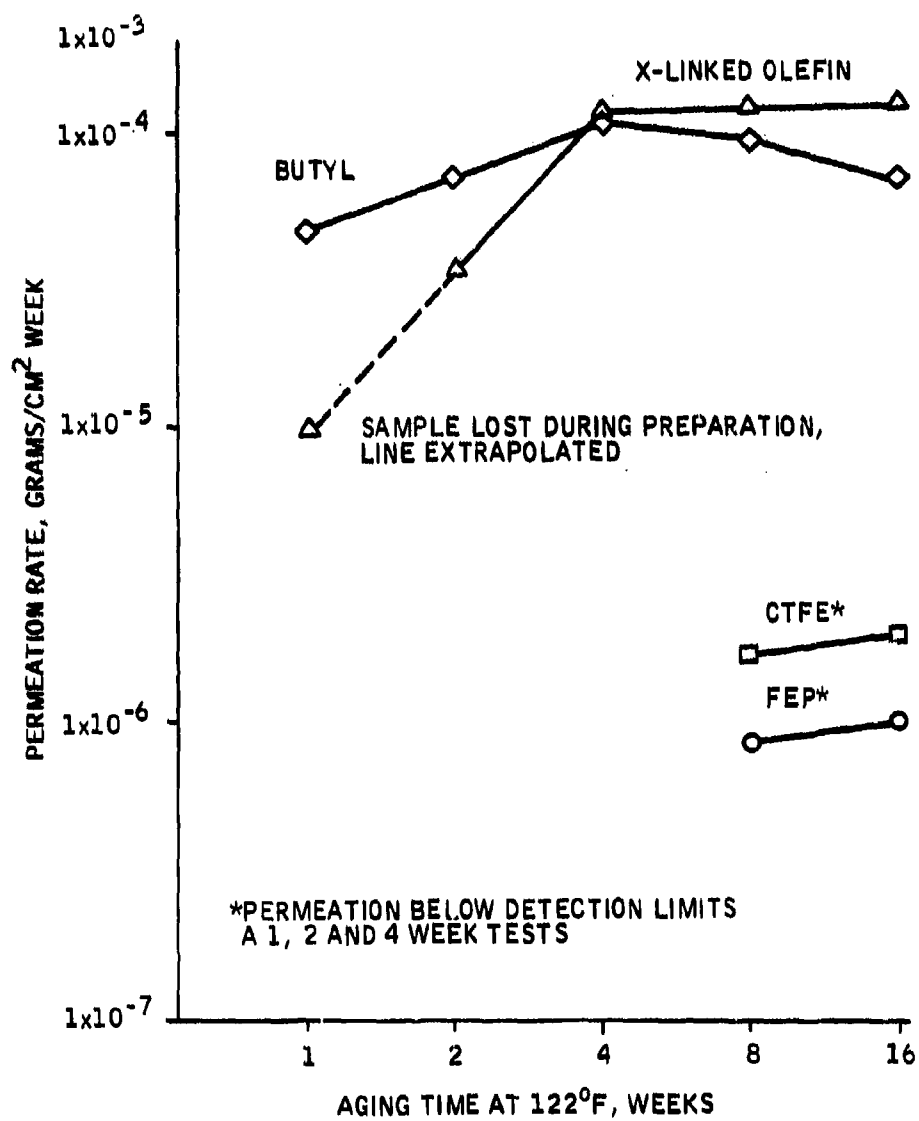


Figure 11. Permeation Rate vs Aging Time

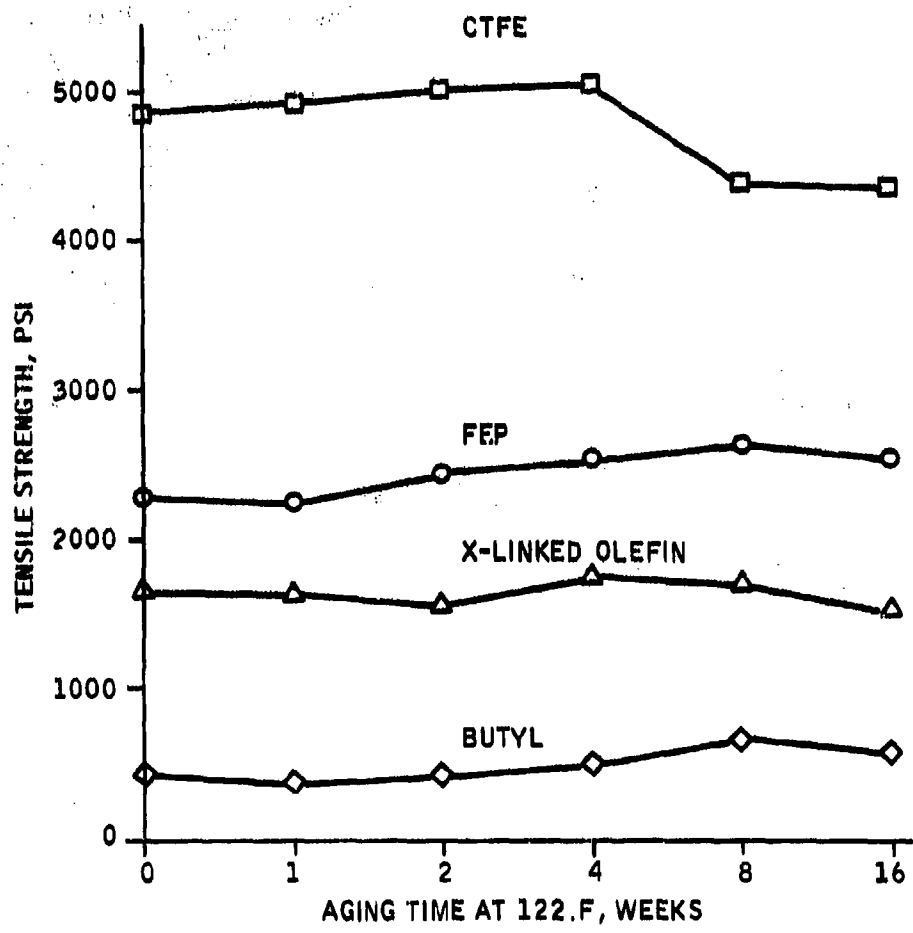


Figure 12. Tensile Strength vs Aging Time

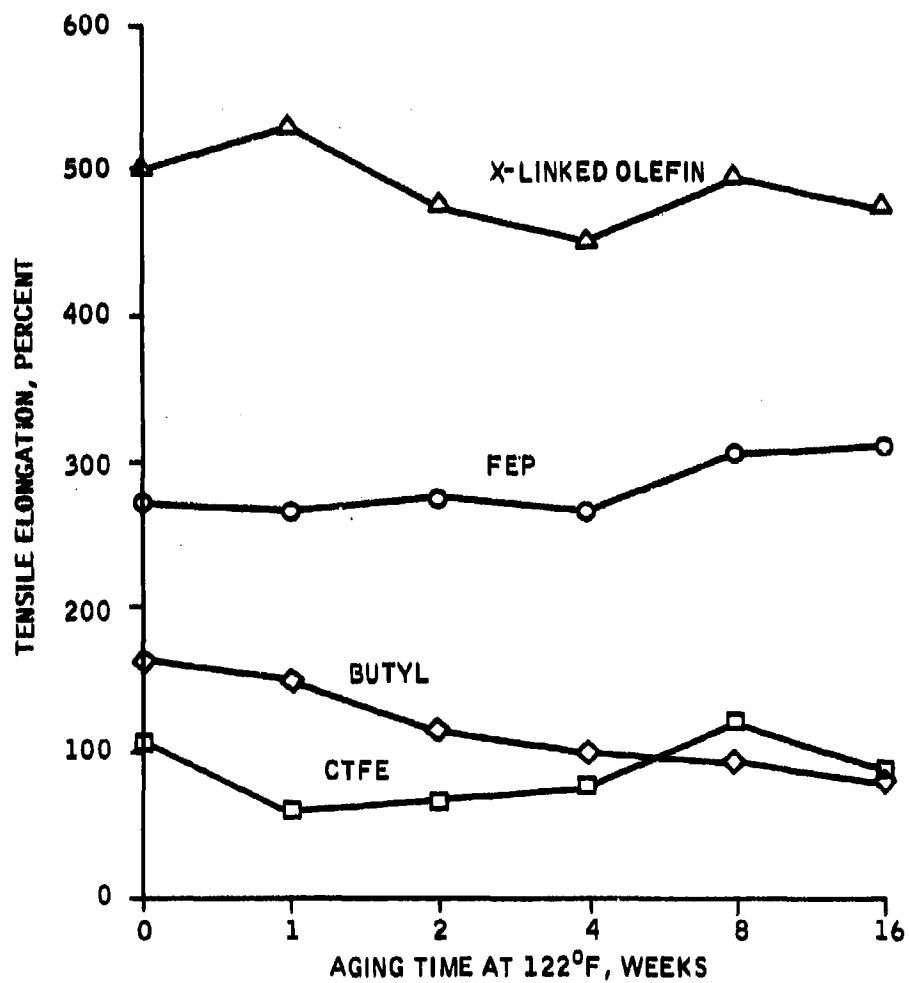


Figure 13. Tensile Elongation vs Aging Time

the specimens was determined via Differential Scanning Calorimetry (DSC). At the end of the second week, when the samples of FEP were found to have softened, hardness checks were added to the test procedure. The results of the DSC tests are shown in Figure 14 and hardness versus exposure time is depicted in Table 7.

Table 7. Effect of Films on Propellant Hardness

Film	Aging Time in Film Envelope (122°F)				
	1 Week	2 Weeks	4 Weeks	8 Weeks	16 Weeks
Teflon FEP	84	40	50	< 28	< 28
Acclar CTFE	99	98.5	95	98	99.5
Butyl LM	99.5	99	99.5	99.5	99.5
X-Linked Olefin RNF1000	99	99	99	99.5	99.5

Control (No aging) = 99.5

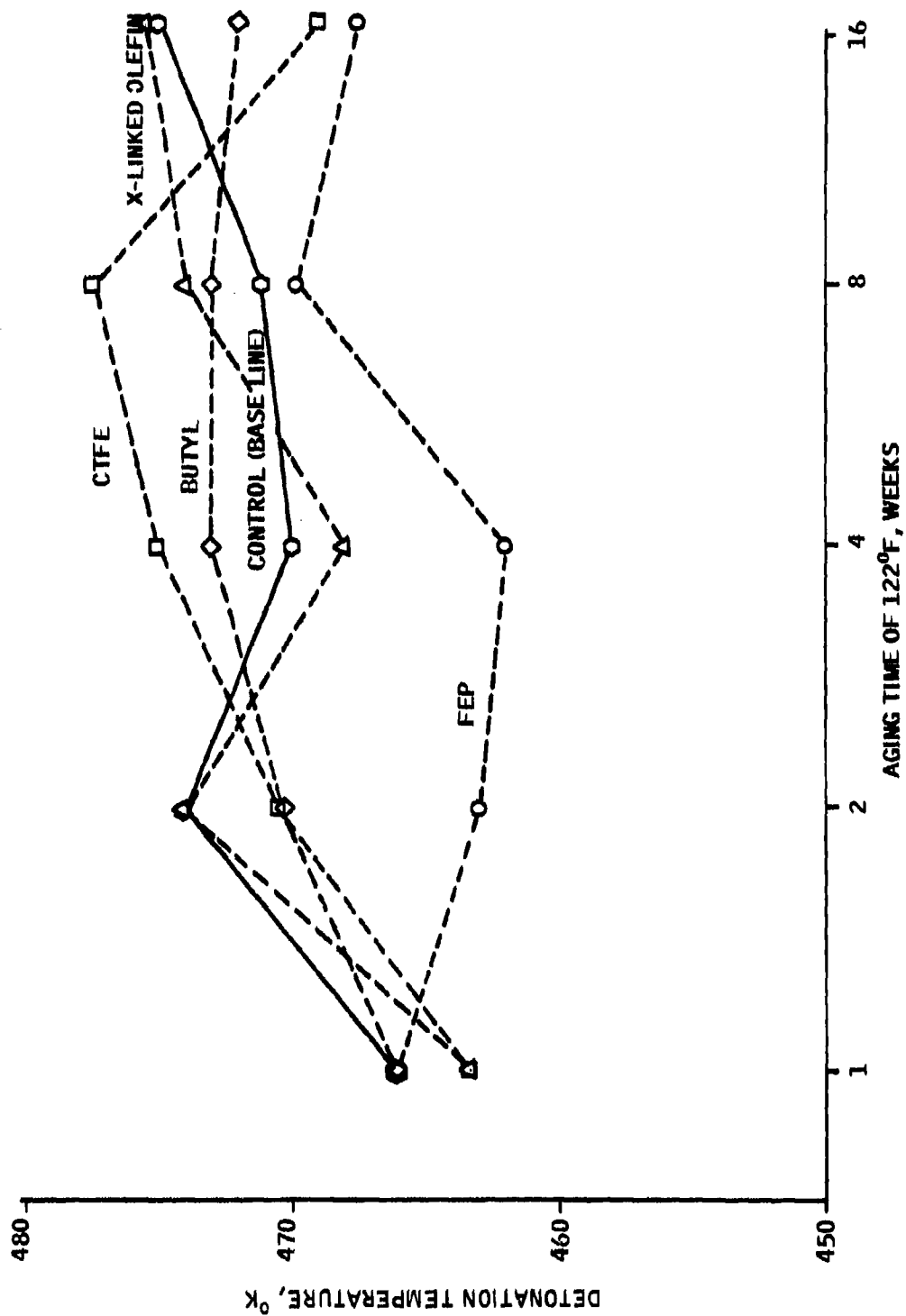


Figure 14. Effect of Aging on M26 Detonation Temperature

SECTION IV CONCLUSIONS

- Of the various parameters used to select polymer candidates, the solubility parameter (δ) provided the best correlation with results obtained from the permeation cell tests.

Permeation was lowest when δ_1 (Nitroglycerin) - δ_2 (barrier) was maximum. Since only four barrier films were tested, and the polarity of the films selected were similar (dielectric constant from 2.1 to 2.5) the effect of polarity cannot be fully evaluated. The glass transition temperature (T_g) of the four polymers tested exhibited no detectable effects on permeation rates.

- The screening tests used provided only limited qualitative data on the film candidates.

Water vapor transmission tests served only to eliminate the most permeable polymers, i.e. Acetal, Mylar, and those in between this range. Immersion in .01M nitroglycerin provided only rough guidelines for polymer selection, probably due to the very low concentration of nitroglycerin. In the end, selection of the four polymers for permeation tests was made with as much weight on process considerations or with screening test data. FEP and CTFE were selected because of their low-moisture transmission rate and weight gain in the immersion tests. One butyl system was selected to include a coating system. The cross-linked polyolefin was selected to provide an additional polymer capable of being heat-shrunk. Both of these processes provide lower cost processing when compared to injection molding, blow molding, or thermo-forming.

- Prolonged permeation tests resulted in a relatively wide range of permeation rates, with the polymers being ranked by increasing permeability as follows: FEP, CTFE, butyl, and cross-linked polyolefin.

The two most permeable polymers (butyl and X-linked olefin) reached equilibrium by the fourth week. These polymers averaged 9.33×10^{-5} and 12.1×10^{-5} grams/square centimeter-week respectively. The two best barriers, FEP and CTFE, had peak values of 1.0×10^{-6} and 2.0×10^{-6} grams/square centimeter-week. However, since nitroglycerin was detectable only at the 8- and 16-week intervals, it is impossible to tell if they had reached equilibrium.

- From a permeation standpoint any of the four films tested might prove acceptable.

Since there is, at present, no clear-cut definition of an acceptable permeation rate, the highest rate of 12.1×10^{-5} grams/square centimeter-week may be satisfactory. We extrapolated the permeation rates to a five inch diameter film in a hypothetical storage environment of 122° F for one year, Table 8. The worst film tested shows a quantity of 0.790 grams spread over the surface of the plug, some of which will certainly have permeated into the plug body. Additional testing would be required before drawing any conclusions on the effects of chemical attack or safety.

Table 8. Calculated Nitroglycerin Permeation (One year storage at 122° F for five-inch diameter film barrier)

Film Type	Nitroglycerin Permeation (grams)
FEP	0.006
CTFE	0.013
Butyl	0.570
Cross-linked Olefin	0.790

- No harmful effects of nitroglycerin vapors on film properties were detected.

The only detectable trend was the slow loss of elongation of the butyl film. Tensile strength and T_g of all films remained relatively constant except for two low T_g readings on the cross-linked olefin at the four- and eight-week tests. These might possibly have been caused by a lack of uniformity of the film. However, the transmission rates were not affected and no problem was created.

- Propellant compatibility test showed FEP to be the only film having interacted with the propellant. It is unknown at this time, however, if this will pose a long-term storage problem.

Propellant samples in direct contact with FEP gradually softened. The softening trend was detectable at the first week and continued throughout the aging period. This effect would be expected if plasticizers were leaving the film and entering the propellant.

According to representatives of DuPont, no material in their film can emanate below 500°F. Hardness checks on some residual samples show the softening effect to be reversible with recovery rates on the order of 40% to 70% per week at room temperature. However, since the quantity of residuals was limited, exact determination of recovery rate would require a re-run of the test series.

Compatibility testing on the propellant exposed to the films was not so conclusive. The wide scatter of M26 detonation temperatures, also observed by Swanson and Madsen (43), preclude any definite statement about compatibility based on DSC testing. In general the CTFE, butyl, and cross-linked olefin exposed samples looked no different than the control samples. The deflagration temperature of samples exposed to FEP were considerably lower than the control at the two- and four-week tests. Additional tests were run via Thermo-gravimetric analysis (TGA). These results showed a slight upward shift (from 140°C to 147°C) of the deflagration temperature when exposed to both Aclar and FEP, see Appendix V. Mass spec. analysis run at 100°C resulted in gas evolution of NO, CO₂ and NO₂, see Appendix VI. The amounts evolved indicated a higher degree of reaction with the Aclar Film when compared with the FEP. This is just the opposite of DSC results. While no hazardous condition is anticipated, the conflicting results of these tests compounded by the capricious nature of the M26 propellant points toward a need for greater systemization and standardization in the area of compatibility testing.

SECTION V RECOMMENDATIONS

The use of FEP to provide an optimal nitroglycerin barrier is recommended. This selection is based on the tests performed. Use of this material, however, should be with the assurance that either the propellant will not come in direct contact with the film or that the softening effect observed is not detrimental to propellant properties. This, of course, would require additional testing. In the absence of such assurances, CTFE is the second choice for barrier material.

Additional testing should be performed since nitroglycerin was not detected until the eighth week of tests with the FEP and CTFE film. Therefore, permeation may not have reached equilibrium. This would mean that extrapolation of the 16 week transmission rates would be slightly low and actual permeated quantities (one year) higher than calculated. Minimum testing time should be 12 months since many ammunition components are stored longer than one year.

Previous tests have defined the quantity of nitroglycerin capable of permeating the barriers selected, efforts should be expended to determine what quantity is acceptable.

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APPENDIX II
BARRIER FILM SCREENING TEST PROCEDURE
N. M. Backstrom

PROCEDURE FOR BAFIL SCREENING TESTS

1.0 Scope

This S. O. P. covers the use of spirits of nitroglycerin as used in the BAFIL screening tests.

2.0 Purpose

To provide safe handling procedures for .01M nitroglycerin (Ng) in the use, storage, and disposal of solutions used in the BAFIL screening tests.

3.0 Reference

Sax Dangerous Properties of Industrial Materials - page 1119.

4.0 Equipment

4.1 Jones and Lamson FC-14 Comparator

4.2 Miscellaneous lab equipment

5.0 Safety

See S. O. P. 1102.

6.0 Security

N/A.

7.0 Procedures

7.1 Barrier film test specimen preparation

7.1.1 Cut two specimens of film selected to 2.8 inch by 0.5 inch size.

7.1.2 Clean specimens with alcohol dampened towels.

7.1.3 Dry specimens at 160°F for two hours.

7.2 Weigh specimens on analytical balance accurately to four decimal places.

7.3 Measure length of specimen using comparator.

7.3.1 Center specimens between two one inch x three inch glass slides.

7.3.2 Mount slides and specimen in comparator.

7.3.3 Adjust instrument to measure length.

7.3.4 Determine length to accuracy of 0.0001 inch.

7.4 Age samples in solutions

7.4.1 Place one specimen of each material in a one pint tin can containing 50 ml. of 0.01 N nitroglycerin in ethanol.

7.4.2 Place one specimen of each material in a one pint can containing only ethanol.

7.4.3 Seal all cans with friction top.

7.4.4 Place all cans containing test specimens in a vented circulating oven at 122°F.

- 7.5 Measure length changes in samples after aging period.
 - 7.5.1 Remove cans containing samples from oven and cool two hours at room temperature.
 - 7.5.2 Place cooled cans near comparator.
 - 7.5.3 Remove specimen from can and insert while wet evenly between two slides.
 - 7.5.4 Make length measurement and return sample to can.
- 7.6 Measure weight changes of specimens after aging period.
 - 7.6.1 Remove specimens from can, wipe dry, and weigh.
 - 7.6.2 Determine percent weight change of each specimen.
 - 7.6.3 Using the percent change of the ethanol specimens as a base, determine the net change of the nitroglycerin-ethanol aged specimens.
- 7.7 Determine volume change of specimens.
 - 7.7.1 By subtraction determine the change in length during the two week aging period of each specimen.
 - 7.7.2 Using Table 1 of ASTM test method D 1480 determine the calculated volume change of each specimen based on the percent length change.
 - 7.7.3 On the basis of the ethanol control samples, determine the net change of the nitroglycerin immersed specimens.

APPENDIX III

CHEMICAL AND METALLURGICAL LABORATORY

STANDARD OPERATING PROCEDURE

D. K. Szidon

PROCEDURE FOR HANDLING SPIRITS OF NITROGLYCERIN

1.0 SCOPE

This SOP covers the use of spirits of nitroglycerin as a standard addition for the polarographic analysis.

2.0 PURPOSE

To provide safe handling procedures for .01M nitroglycerin (Ng) in the use, storage, and disposal of solutions in cells for polarographic analysis.

3.0 REFERENCE

SAX - Dangerous Properties of Industrial Materials, page 1119.

4.0 EQUIPMENT

4.1 PAR polarographic analyzer Model 174 and associated accessories

4.2 Standard sample preparation equipment

5.0 SAFETY

5.1 Care will be exercised to prevent spills of the spirits and evaporation of solvent. Residue after evaporation is nitroglycerin, a high explosive, which is very sensitive to friction and shock. Fumes are toxic when heated, and it reacts vigorously with oxidants.

5.2 Spills will be neutralized by scrubbing and flushing with a solution consisting of:

1.5 liter H_2O

3.5 liter denatured alcohol

1 liter acetone

500 grams sodium sulphite (60% commercial)

NaOH solution 1M, (instead of the above mixture)

5.3 Skin contact should be avoided but in such instances scrub with soap and flush well.

5.4 Avoid breathing fumes as serious headache and dizziness may ensue.

5.5 Dispose of all sample material in solvent waste container.

5.6 Clean and rinse all glassware with methyl alcohol and deionized water. Flush sink with tap water.

5.7 Any heating of sample material will be in a water bath in a hood.

5.8 Spirits of nitroglycerin standard will be stored in a tightly sealed glass bottle kept in the solvent cabinet.

5.9 All incoming materials for analysis will be stored in a cool dry place (Ng barrier film test cells).

6.0 SECURITY

Not applicable

7.0 PROCEDURES

7.1 Barrier film test cell preparation

- 7.1.1 Wash cells with H_2O , acetone, and MeOH
- 7.1.2 Bake cells for three hours minimum at $100^\circ C$
- 7.1.3 Repeat wash of 7.1.1, drying of 7.1.2
- 7.1.4 Place cells in dry room ($RH < 5\%$)

7.2 Silica gel preparation (absorption media for NGO)

- 7.2.1 Dry silica gel at $100^\circ C$
- 7.2.2 Seal in glass container
- 7.2.3 Place container in Dry Room ($RH < 5\%$)

7.3 Propellant Sample Preparation

- 7.3.1 Cut into discs $.080 \pm .015$
- 7.3.2 Place in glass container in Dry Room

7.4 Loading Test Cell

- 7.4.1 All work to be conducted in Dry Room
- 7.4.2 Label and/or record all cell numbers, color, film type, test period, etc.
- 7.4.3 Weigh out 2.000 g propellant and place in top portion of cell with screen. Record propellant weight.
- 7.4.4 Weigh out 1.500 g of dried silica gel and place in bottom portion of cell (cup). Record weight of cup, cup + silica gel, and net weight gain of cell as silica gel.
- 7.4.5 Assemble cells and place in test oven at $122^\circ F$ for specified time period.

7.5 Test Cell Analysis

- 7.5.1 Weigh and record weight of test cell cup before and after transfer of silica gel to erlenmeyer
- 7.5.2 Wash test cell cup with 10 ml of 50/50 EtOH/ H_2O in 2 or 3 aliquot portions. Transfer washings to erlenmeyer with the silica gel
- 7.5.3 Add additional 5ml of 50/50 EtOH/ H_2O to erlenmeyer, heat in water bath one hour, and then let cool.

7.6 Polarographic Analysis

- 7.6.1 Place 10ml of INR_4NBr in cell with 10ml of extract from erlenmeyer and bubble with N_2 for 20 minutes.
- 7.6.2 Analyze polarographically with differential pulse mode at -1.42 volts versus Saturated Calomel Electrode.
- 7.6.3 Make addition with Ng standard of 0.01001M
- 7.6.4 Measure peak heights and calculate Ng in grams
- 7.6.5 Report quantity of Ng found

APPENDIX IV
EXPLOSIVE COMPATIBILITY TEST PROCEDURE

N. M. Backstrom

J. L. Madsen

EXPLOSIVE COMPATABILITY TEST PROCEDURE

1.0 SCOPE

This SOP covers the use of M26 propellant as used in the Bafil Explosive Computability Test.

2.0 Purpose

To provide safe handling procedure of M26 propellant during its use, storage, and disposal for the Bafil Computability Tests.

3.0 Reference

- 3.1 Sax. Properties of Dangerous Industrial Materials, page 1119
- 3.2 Honeywell SOP 348
- 3.3 Honeywell DOP 3383

4.0 Safety and Handling

- 4.1 The propellant must not be exposed or subjected to excessive heat, friction, electrical discharge or open flame.
- 4.2 Safety glasses with side shields will be worn during handling, machining and testing.
- 4.3 The tested propellant is properly boxed, identified and delivered to explosives lab for disposal.

5.0 Equipment

- 5.1 Wallace Micro-indentation tester.
- 5.2 Perkin Elmer. DSC 1B Differential Scanning Calorimeter.

6.0 M26 Compatibility test procedure.

6.1 Sample preparation.

- 6.1.1 Using a sharp heavy pocket knife, cut a $0.060'' \pm 0.015''$ slice from the M26 pellet ($0.5'' \times 1.5''$ approx.).
- 6.1.2 Using the pocket knife, quarter the M26 slice into 4 pie shaped pieces.
- 6.1.3 Place one M26 test piece (6.1.2) between two (one inch) squares of clean (alcohol wiped) film barrier material and secure the resulting sandwich with 3 or 4 wire staples.

6.2 Aging procedure.

- 6.2.1 Place specimen(s) in clean 1/4 pt. glass jar. Seal with cover.
- 6.2.2 Place jar and specimen(s) into 122°F oven.

6.3 Hardness test procedure as a M26 compatability criterion.

- 6.3.1 Determine hardness of M26 test pieces before and after aging period using the Wallace Micro Indentation tester.

6.4 DSC Testing procedure.

- 6.4.1 Cut approximately 5 Mg sample from test specimen and crimp into standard DSC sample pan.
- 6.4.2 Scan sample as per standard DSC procedures from room temperature to decomposition at a heating rate of 10°C/MIN and a range setting of 32 millical/°C sec.
- 6.4.3 Record the peak of the decomposition exotherm as the decomposition temperature.

APPENDIX V

TGA EXAMINATION OF M26 WITH FEP AND ACLAR FILMS

COPY LIST:
LIST A



GOVERNMENT AND AERONAUTICAL
PRODUCTS DIVISION

REPORT NO. T1433

DEV. NO. W9416-AC-0003-2643

DATE 4 October 1974

PAGE 1 OF

ISSUED BY: GAPD PLASTICS LABORATORY

TITLE: TGA examination of M26 with FFP and ACLAR films.

OBJECT:

Determine if any gross incompatibility exists between M26 propellant and REP and ACLAR films.

MATERIALS TESTED:

M26 - NOS Indianhead
FFP film - Lab stock
ACLAR film - Lab stock

CONCLUSIONS:

TGA results indicate that FFP and ACLAR films will not cause M26 to become unstable or hazardous.

PROCEDURE AND RESULTS:

Samples of M26, FFP and ACLAR were run on the TGA at 2.5°C/min. Neither the FFP or ACLAR exhibited any weight loss below 200°C. The M26 gradually started to lose weight above 75°C until it deflagrated at 140°C. Samples of M26 were run with FFP and ACLAR also at 2.5°C/min. Both these runs were similar to the control M26 run with the exception both deflagrated at 147°C.

Because neither FFP or ACLAR caused M26 to decompose more readily or deflagrate at a lower temperature than the control sample, it can be assumed that neither of these materials are grossly incompatible or hazardous when used in conjunction with M26 propellant.

KEYWORDS:

Thermal

ATTACHMENTS:

Figs 1-V

DATA BOOK NO. PAGE			
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REQUESTED BY	DATE	WRITTEN BY	
N. Gregornik	9/15/74	James Madsen	
DEPARTMENT		APPROVED	
PLASTICS LAB.			

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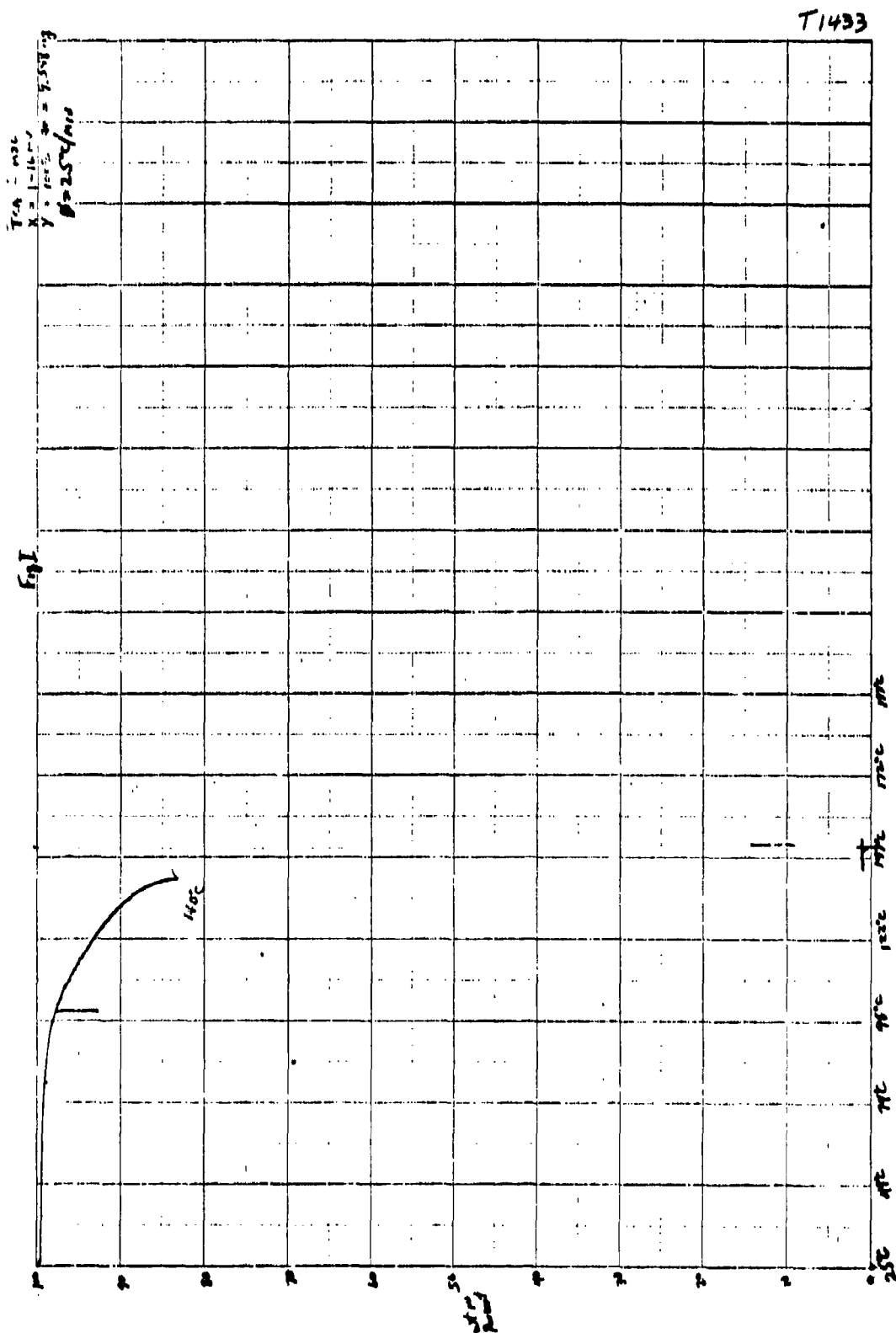


Figure 1

FEP File
 $g = 2.5 \times 10^{-4}$
 $R_1 = 1.000$
 $R_2 = 1.002$

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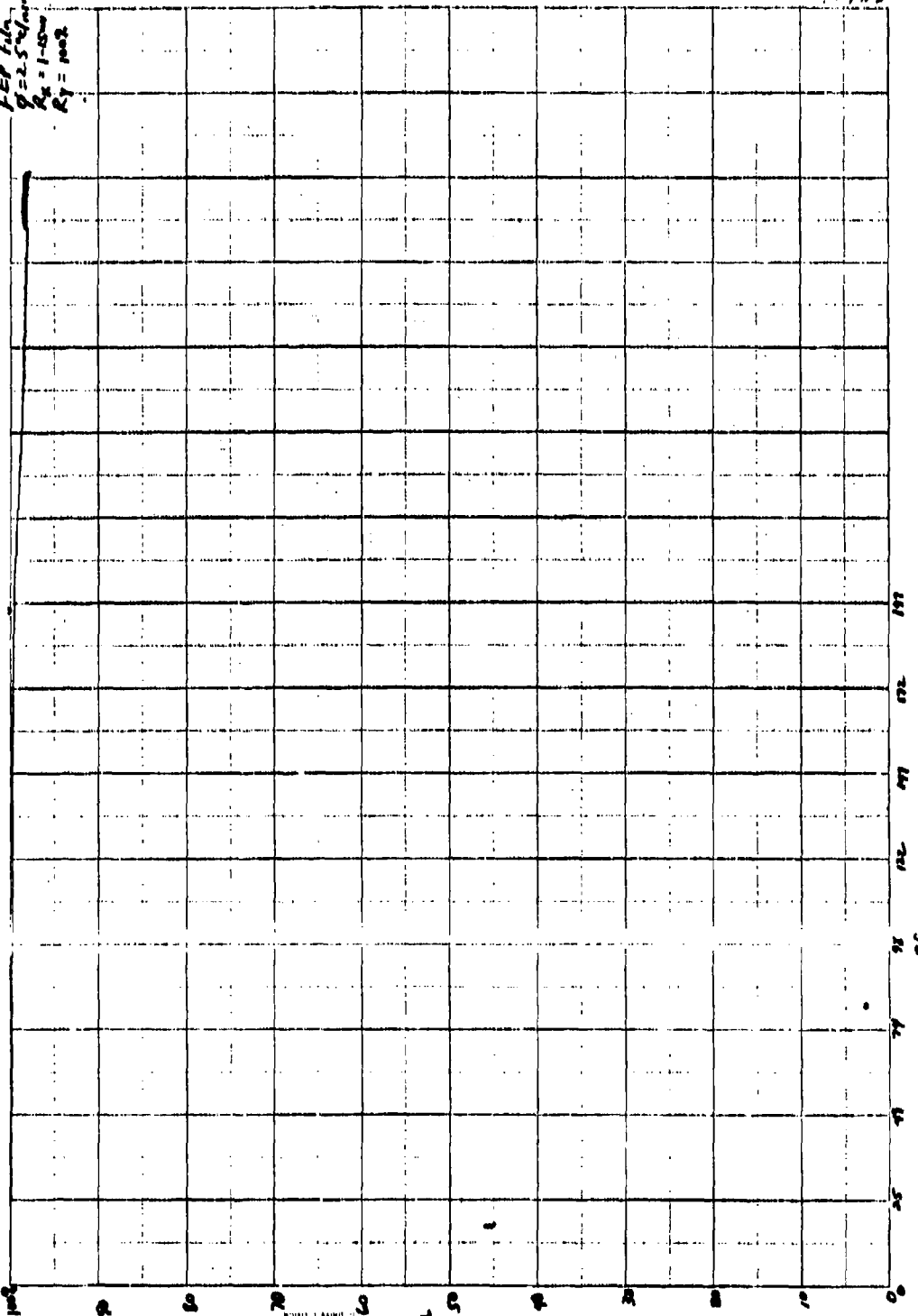


Figure 2

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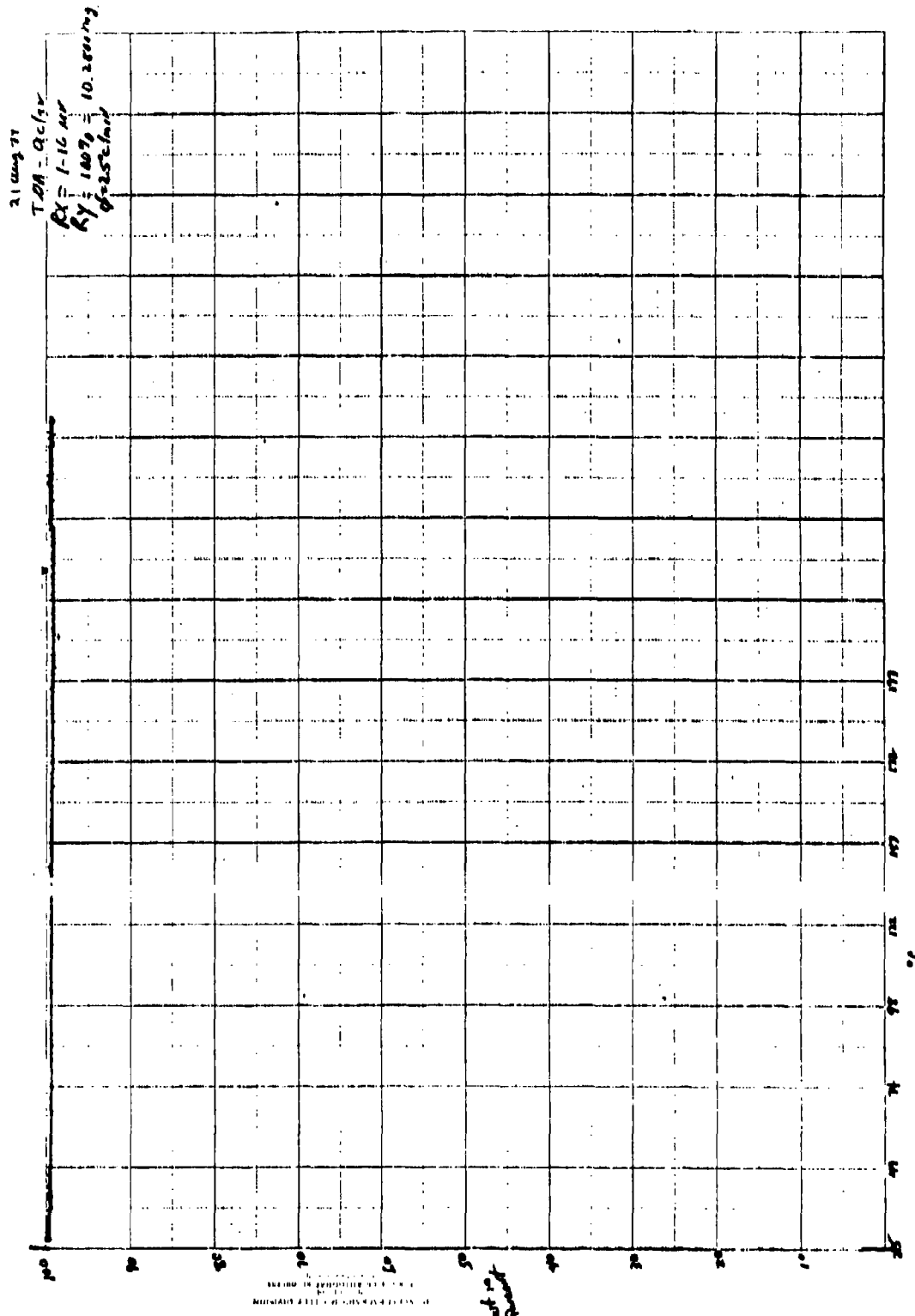


Figure 3

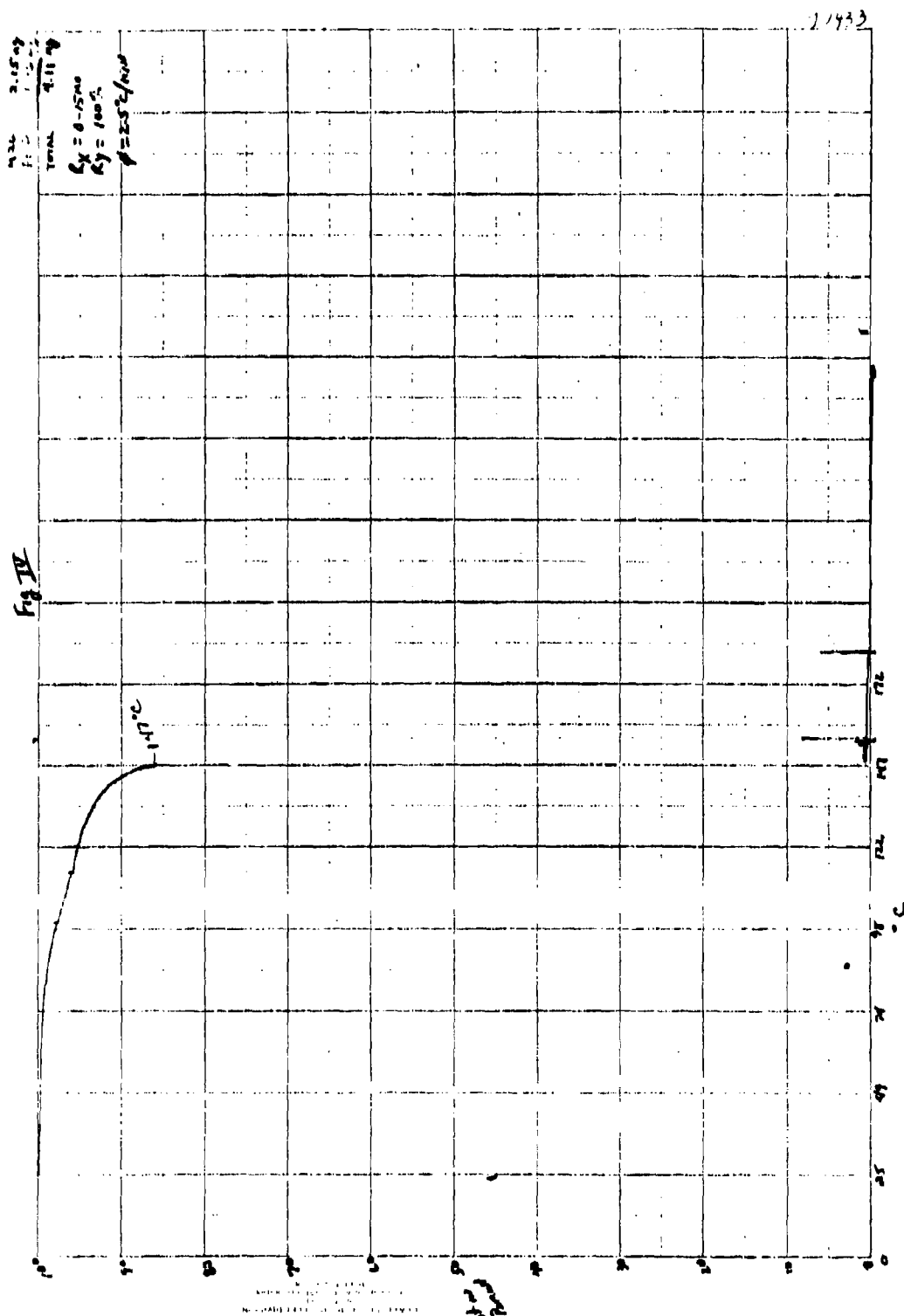


Figure 4

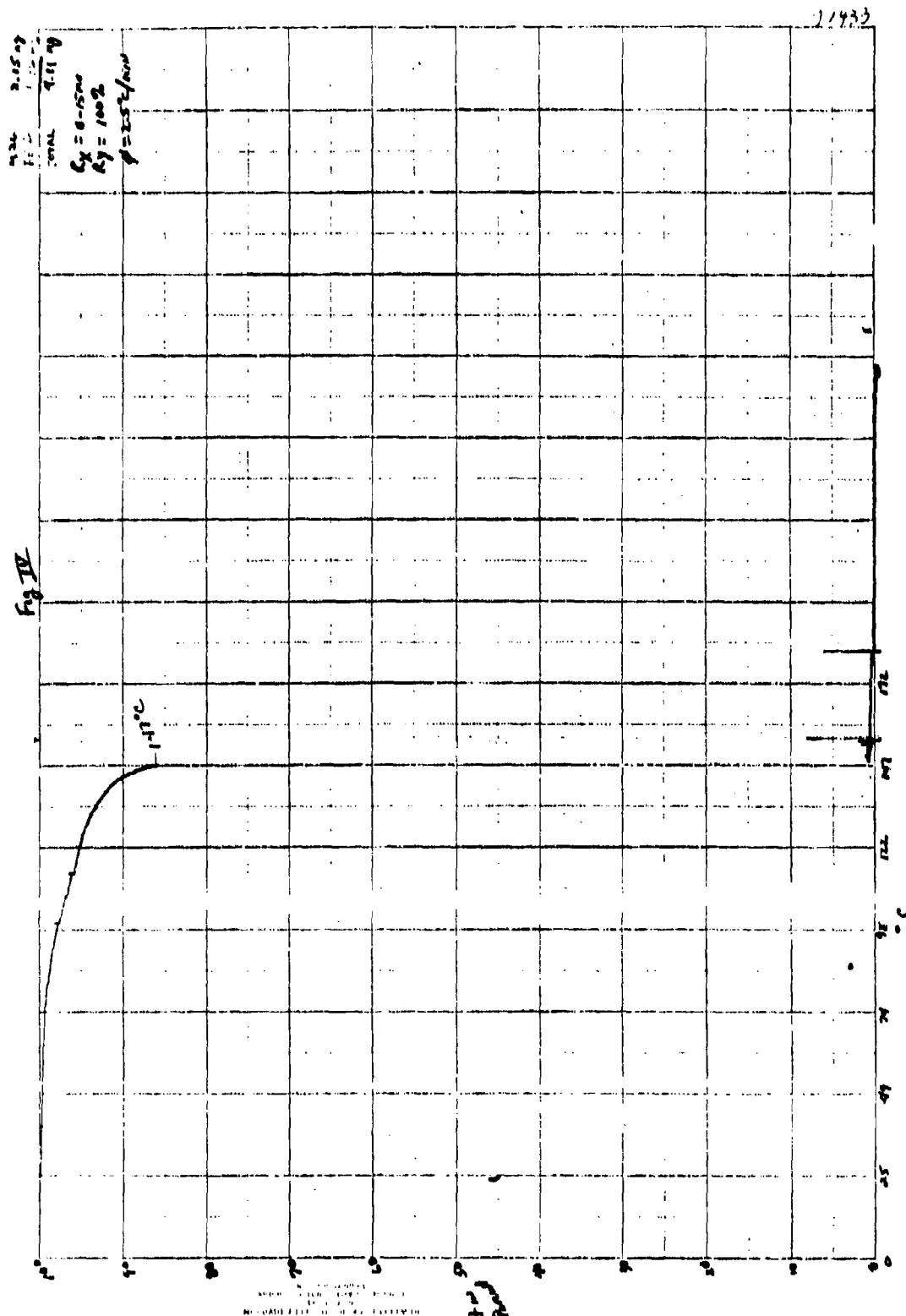


Figure 4

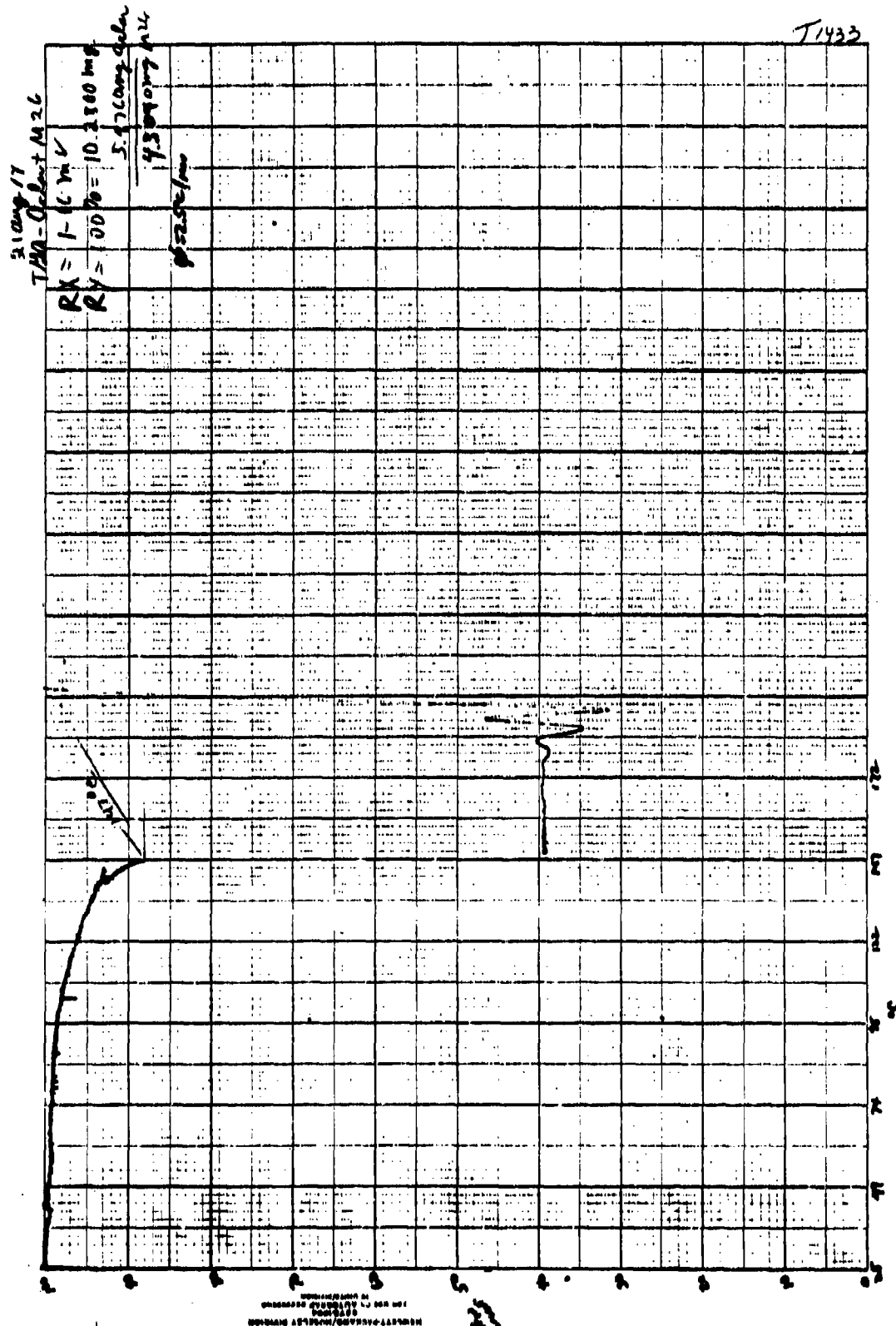


Figure 5

APPENDIX VI
MASS SPECTRAL ANALYSES OF PROPELLANT-PLASTIC SAMPLES

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GOVERNMENT OF CANADA
PRODUCTS DIVISION

REPORT NO. 6806
DEV. NO. W9416-AC-0002-2643

ENGINEERING TEST REPORT

DATE October 4, 1974

PAGE 1 OF 2

ISSUED BY: G&APD CHEMICAL LABORATORY

SUBJECT: Mass Spectral Analyses of Propellant-Plastic Samples

BACKGROUND:

Two samples of propellant wrapped in plastic and one sample of propellant alone were submitted for mass spectral compatibility analysis.

The propellant (M26) was a double based propellant containing 25% nitroglycerin. The plastic samples were Teflon FEP and Aclar.

ACTION REQUESTED:

Analyze gases emitted, with mass spec, when propellant and plastic were heated together.

EXPERIMENTAL:

The samples were placed in Fischer-Porter tubes which were evacuated and backfilled with Neon gas to a pressure of 20 mmHg. These tubes were then heated at 100°C for 24 hours. When cooled, the gas phase contents were analyzed on the mass spectrometer.

RESULTS:

A ten scan average of the propellant-Teflon sample yielded the following results. All peaks were normalized to Neon at AMU 20.

AMU	Species	Relative Abundance
16	O	5
18	H ₂ O	160
20	Ne	100
28	N ₂	606
30	NO	17
32	O ₂	196
44	CO ₂	38
46	NO ₂	11

ATTACHMENTS:

DATA BOOK NO. PAGE

REQUESTED BY
Norm Gregornik

DATE
10/4/74

WRITTEN BY
Roger Keller

DEPARTMENT
Plastics Lab

APPROVED

10/4/74 1c
HE-44A REV. 9/71

John H. Fossum
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RESULTS: (continued)

A ten scan average of the propellant-Aclar sample gave the following results normalized to Neon:

<u>AMU</u>	<u>Species</u>	<u>Relative Abundance</u>
16	O	25
18	H ₂ O	258
20	Ne	100
28	N ₂	594
30	NO	180
32	O ₂	6
44	CO ₂	419
46	NO ₂	3

The propellant alone gave the following results:

<u>AMU</u>	<u>Species</u>	<u>Relative Abundance</u>
18	H ₂ O	42
20	Ne	100
28	N ₂	178
30	NO	3
32	O ₂	25
44	CO ₂	6
46	NO ₂	6

CONCLUSIONS:

Relative to the control sample (propellant alone) both plastic samples gave off considerable amounts of NO gas, especially the Aclar sample. The same pattern is observed with CO₂ and NO₂. This would indicate some reactivity between the propellant and the plastics with the Aclar being somewhat more reactive than the Teflon FEP.

CONCLUSIONS:

The plastics, Teflon FEP and Aclar, are halogenated hydrocarbons which are generally considered to be chemically inert. The propellant is 75% nitrocellulose and 25% nitroglycerin. The source of nitrogen oxide gases is the nitro groups of the propellant compounds. Nitrogen is also a degradation product of organic nitro compound breakdown. The increased CO_2 and water associated with the Aclar sample may also be a breakdown product.